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FEASIBILITY INVESTIGATION OF CHEMICALLY
SPRAYED THIN FILM PHOTOVOLTAIC CONVERTERS

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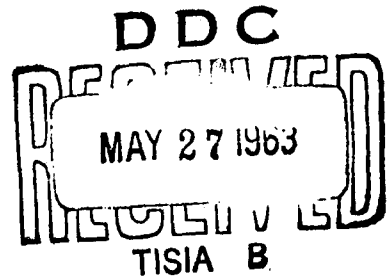
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Aeronautical Systems Division

Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared by The National Cash Register Company covering work done under Air Force Contract 33(657)-7919; however, this report is being published and distributed prior to Air Force review. The publication of this report, therefore, does not constitute approval by the Air Force of the findings or conclusions contained herein. It is published for the exchange and stimulation of ideas.

The contract was initiated by the Flight Accessories Laboratory of the Flight Vehicle Power Branch as Task 817301, Project 8173, "Feasibility Investigation of Chemically Sprayed Thin Film Photovoltaic Converters".

The work was administered under the direction of the Aeronautical Systems Division, Wright-Patterson Air Force Base, with Mr. L. D. Massie as Project Engineer.

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I. INTRODUCTION

The objective of this work is to conduct the necessary applied research to obtain the experimental data which will demonstrate the feasibility of fabricating a thin film photovoltaic converter, using a chemical spray process, and if feasibility is shown, to extend the research to the problems of fabricating a large area thin film flexible solar cell array.

Previous quarterly reports have covered the research efforts in the areas of instrumentation; deposition of transparent conductors; optical, electrical, and physical characteristics of CdS thin films (.5-1.0 μ range); fabrication of a thin film photovoltaic converter using a $\text{SnO}_x\text{-CdS-Cu}_x\text{S}_y\text{-metal}$ system where the cadmium sulfide layer is approximately 0.6 μ thick, the copper sulfide is approximately .08 μ thick, and both are deposited with the same chemical spray technique.

Technical Documentary Report No. ASD-TDR-63-223, Part 1 of March 1963 summarizes the work done under this contract from 1 February 1962 through 31 January 1963.

This report covers the work done since 31 January 1963 in translating the techniques developed in fabricating cells on a glass substrate with a transparent conductor (back wall configuration) to fabricating cells using a metal foil as a substrate (front wall).

In addition to the fabrication efforts with metal substrates, the report covers the work done in investigating the nature of the junction formed between a copper sulfide film and an n-type material; and the setting-up of equipment to measure the Hall coefficient of high resistivity thin films.

The work done in studying the formation of a junction using chemically sprayed copper sulfide and single crystals of CdS, CdTe, and Si is also discussed in this report.

II. CELL FABRICATION

A. Substrate

The choice of a material to use as the flexible substrate for a large area thin film array, molybdenum foil, was initially made on the basis of thermal stability and close match of thermal expansion coefficient (CdS is $4.2 \times 10^{-6}/\text{C}^\circ$ and Mo is $4.9 \times 10^{-6}/\text{C}^\circ$). This thermal expansion match between Mo and CdS is especially important when a thick (10-100 μ) layer of CdS is being deposited and particularly where adherence during heat cycling is a problem. The chemical spray process is, perhaps uniquely, capable of depositing a very well-adhering film in the 0-1.5 μ range on every substrate (glass, quartz, mica, Mo, W, Pt, Ni, etc) used to date if the substrate is capable of withstanding deposition temperatures; however, as pointed out in previous interim reports ⁽¹⁾ unwanted reactions occur with the substrate, such as forming PtS with Pt and MoO₃ when Mo is being used as the substrate film. This unwanted reaction is coupled with the additional problem of poor film crystallinity when metal is used. When an oxide forms (the usual case) an intervening insulating layer results which seriously hinders the subsequent formation of an efficient photovoltaic cell.

Metal foils other than Mo, together with several different metals plated on a like foil, have been tried with the same result of an unwanted reaction with the substrate. This unwanted reaction with the substrate can be avoided by lowering the substrate temperature, but as found even

with glass substrates, as the deposition temperature is lowered the crystallinity of the film changes such that the resulting film is completely amorphous to x-ray. It has been shown, as previously reported (2), that good film crystallinity (crystallites at least greater than .1 μ in size) is needed to observe any appreciable photovoltaic effect. Consequently, lowering the deposition temperature merely trades one problem for another. Heat treatments of films which were initially amorphous to x-ray have never resulted in any appreciable crystal growth. It has been found that a crystallinity to an extent at least detectable by x-ray (minimum crystallite size about 500 \AA) is needed before heat treatment

The problem of obtaining good crystallinity on a metal substrate has been solved by depositing first an amorphous CdS layer at low temperature with a subsequent deposition of a second CdS layer at a much higher temperature. This dual deposition has resulted in good crystallinity in the final film-metal structure. While the initial deposition of an amorphous film forms a glass-like surface which appears to be very conducive to a subsequent deposition of a crystalline film, it does not prevent some reaction with the metal from being reflected upwards and through the second film. These unwanted reactions cause the top surface to lack smoothness and uniformity and appear to be the reason that current collection has been difficult with a broad area contact on the front wall Mo foil CdS cells.

Problems encountered in the attempts to deposit crystalline CdS films on metal foils have led to a re-evaluation of the substrate to be considered usable in a high power-to-weight ratio photovoltaic cell. Experience to date indicates that crystallinity in the CdS layer on a

metal substrate can only be obtained when a thin film of amorphous CdS is deposited prior to the deposition of a "normal" CdS film. The necessity for depositing two distinctly different CdS films increases the problems of uniformity and reproducibility of the properties needed for efficient solar cells.

Early CdS cells were deposited on SnO_x coated glass substrates. The conversion efficiency of these cells is considerable but the normal glass thickness of about 1/16" results in a very low power-to-weight ratio. By employing SnO_x coated Microsheet ⁽³⁾ glass substrate a gross change can be made in the weight of the cell.

Discussed here are some of the considerations concerning the power-to-weight ratio for a completed 3" x 3" cell on 5 mil microsheet glass operating in the back wall configuration at a conversion efficiency of 3.0% under 140 milliwatts/cm² incident irradiation (see Figure 1).

Weight for 3" x 3" area

5 mil thick glass substrate	1.950 gms
SnO_x Transparent Conductor (6000 Å)	.024 gms
Cadmium Sulfide 5000 Å thick	.014 gms
Copper Sulfide (Cu_xS_y) 1000 Å thick	.004 gms
Copper Current Collector 1000 Å thick	.005 gms
Indium Contact to SnO_x 1/16" wide around periphery	.048 gms
1 mil mylar + adhesive (bonded to cell for protection)	.325 gms
	<hr/> 2.370 gms

Allowing 1/8" around the periphery of the cell for inactive area to make contact to the SnO_x and to isolate the copper current collector

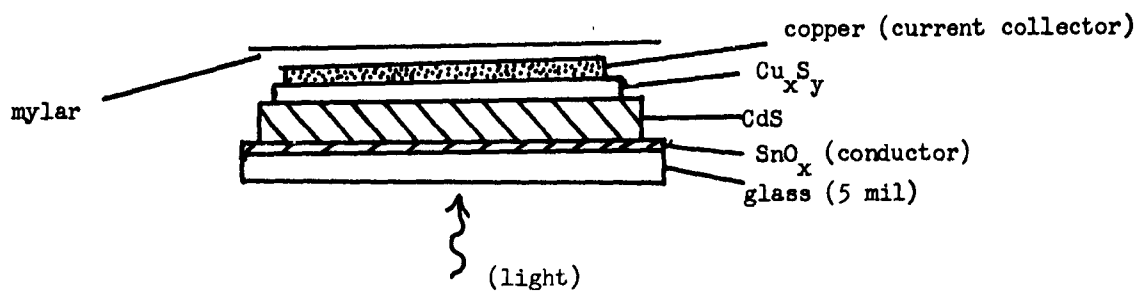


FIGURE 1

Back Wall CdS Cell on Glass Substrate

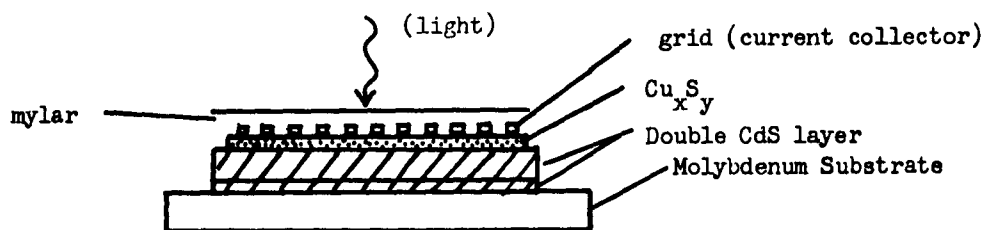


FIGURE 2

Front Wall CdS Cell on Metal Substrate

electrode leaves an active area of 7.55 in^2 or about 48 cm^2 .

Allowing an incident energy of 140 milliwatts per cm^2 gives a total usable energy of 6.720 watts. Since we are assuming for this evaluation a conversion efficiency of just 3%, then the usable power from such a cell would be 0.202 watts. The total cell weight is 2.37 gms or 5.25×10^{-3} lbs. This results in a power-to-weight ratio of 38 watts/lb.

Considerations for determining the power-to-weight ratio for a 3" x 3" area, 3% efficient front wall cell on 2 mil thick molybdenum foil (see Figure 2):

	<u>Weight for 3" x 3" area</u>
2 mil thick molybdenum foil	2.781 gms
CdS Amorphous (2000 Å°)	.0061 gms
CdS Crystalline (5000 Å°)	.014 gms
Cu_xS_y grid (20 lines/in)(1 mil thick)(48 cm^2)	.044 gms
1 mil mylar + adhesive (electrode bond)	<u>.325 gms</u>
	3.174 gms

Assuming the same active area for the CdS cell on molybdenum as on the glass gives the same output energy of 0.202 watts. Since the weight of the cell is 7.5×10^{-3} lbs the power-to-weight ratio is 26.9 watts/lb.

From the above considerations it is suggested that an advantage in the power-to-weight ratio can be obtained for cells of equal energy if advantage is made of the low density and transparency inherent in glass. In the examples cited above an increase of 41 percent in power-to-weight ratio was gained by using a 5 mil glass substrate in place of a 2 mil molybdenum foil.

B. Film Deposition

The deposition of crystalline films, where the films are to be used in a direction perpendicular to their plane, is complicated if the surface of the substrate has any imperfections which might offer favorable nucleation sites to the growth of abnormally large crystallites which in turn disrupt the upper surface smoothness of the film which is being deposited.

Since this sort of growth characteristic is common to films deposited by the chemical spray process it appeared necessary to investigate the polishing of the Mo which was initially chosen as the "flexible" substrate. It has been found that, with a few minor changes, the electrolytic polishing techniques for molybdenum metallographic specimens reported by Coons ⁽⁴⁾ can be utilized on the small scale required for our purposes.

The experimental set-up being utilized is shown in Figure 3.

The optimum voltage required is in the range of 10 to 11 volts while the current is dependent on the following:

- (a) Surface area of the metal to be polished
- (b) Volume of electrolyte being used
- (c) Temperature of the electrolyte, current being directly proportional to the temperature (if the temperature is increased, the resistance of the electrolyte decreases with a resultant increase in current for constant applied voltage).

In the indicated experimental set-up, the initial current during the polishing process is 32 to 30 amps; after a few seconds this drops to a plateau in the range of 27 to 22 amps, the plateau attained being

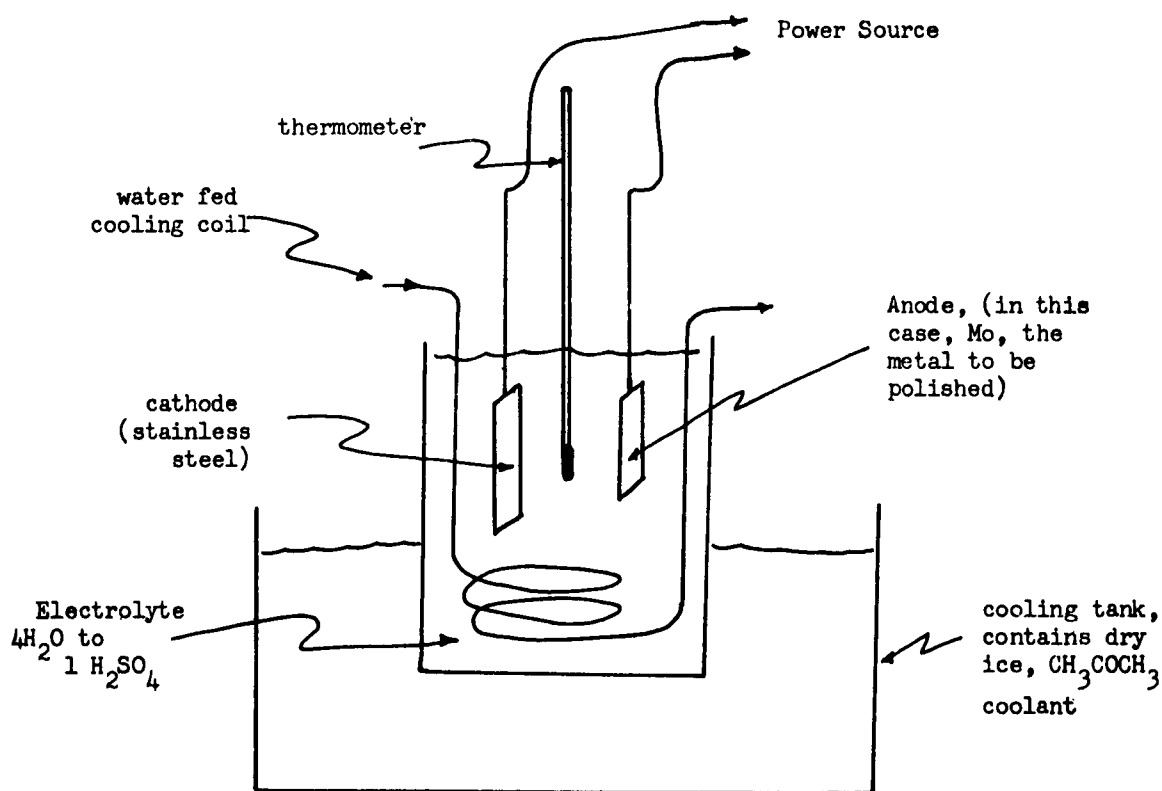


FIGURE 3

Experimental Apparatus for Electropolishing Mo

dependent on the surface area of the sample being polished. The length of time for which a sample is kept in the electrolyte during the polishing procedure is in the range of 15 to 45 seconds, the specific time also being dependent on the surface area of the sample being polished. If, during the polishing procedure, a sample is kept in the electrolyte for an over-extended length of time, pitting and streaking will occur and eventually the continuing chemical reactions occurring at the surface of the metal sample will cause gross dissolution of the metal. Generally, it can be stated that the time of treatment necessary for the polishing process is inversely proportional to the current density used. The temperature range required to maintain the aforementioned current ranges is 45° to 55°F. As noted previously, temperature and current are directly proportional, hence, a decrease in temperature will bring about a decrease in current, consequently longer polishing time is required. Polished Mo foil substrates do permit the deposition of amorphous films which have greater optical clarity and smoothness than those obtained with just "cleaned" foil. This improvement of the physical characteristics due to the polishing has not yet improved the final cell. however.

An investigation of possible changes which might be made in the deposition process to improve or change the film crystallinity has resulted in the deposition of a film of CdS which when heat treated showed, for the first time with a chemically spray deposited film, a major change in the direction of preferred orientation. This change in orientation shown in the x-ray diffractograph as Figures 4 and 5 is similar to that which takes place with the recrystallization process shown by Van Cakenburghe and Giles ⁽⁵⁾, but in this case the transition took place

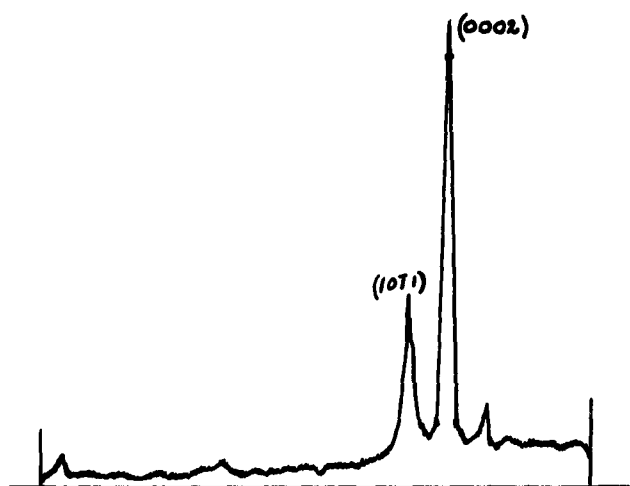


FIGURE 4

X-ray Diffractograph of a CdS Film

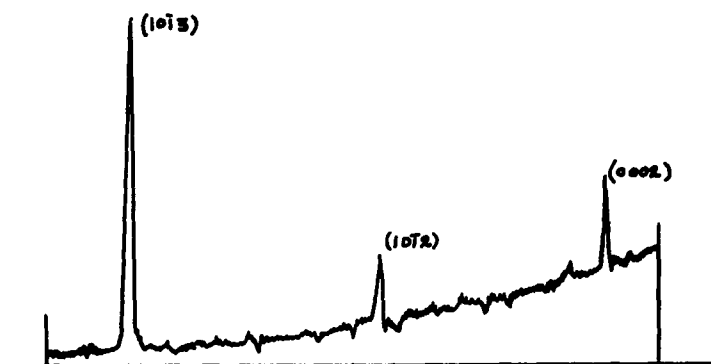


FIGURE 5

X-ray Diffractograph of a Heat Treated CdS Film

without seeding and the crystallites did not grow to the point that any change could be detected optically using polarized light. This deposition change has not as yet given any measurable improvement in CdS films deposited on metal.

C. Test Results

Testing of the front wall cells which have been fabricated to date has been accomplished through the use of a pneumatic set fixture such as that designed for this same purpose at the Harshaw Chemical Company. (6) This fixture provides convenient testing of the same cell using a variety of metal screens or the testing of individual areas of the same cell.

Table 1 gives the test results for eight front wall cells using polished and unpolished Mo. Comparison of the values using a mesh as the collector and using a point probe as the collector shows quite clearly that the current did not increase proportionately to collector area and the open circuit voltage decreased. Had the current collection been done using either silver paint or evaporated metal the values for one cm² areas would, due to pinhole shorting, have been much lower.

The I-V curve of a cell made on metal indicates that only a slight rectification is present and as the area of the front current collector is increased the front to back ratio decreases rapidly to almost one.

The spectral response of a typical metal-base front-wall cell is shown in Figure 6.

Table 2 gives the values measured for a number of cells (back wall on glass) which were fabricated about six months ago. These cells were given no encapsulation or special treatment and were stored in the normal laboratory environment (40-50% relative humidity at 72°F). Comparison

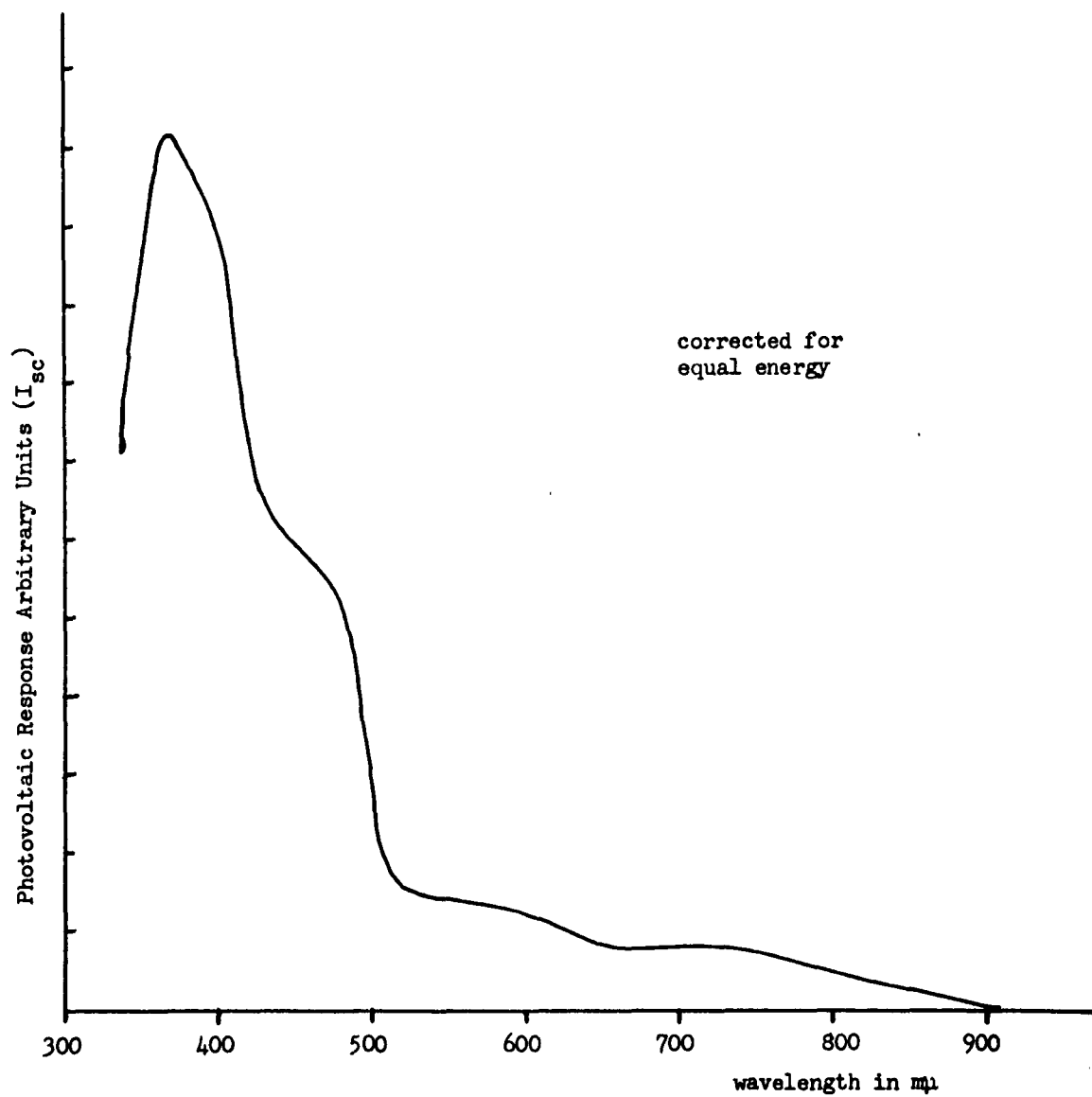


FIGURE 6
Spectral Response of a Front Wall CdS Cell on Mo Foil

of the values measured at the time of fabrication and those values measured months later show the stability which is possible.

<u>Cell #</u>	<u>Substrate Surface</u>	<u>Point Probe</u>		<u>1/2 cm²</u>	
		<u>V_{oc}</u>	<u>I_{sc}</u>	<u>V_{oc}</u>	<u>I_{sc}</u>
287 C	polished	.35 V	.2 ma	.22 V	2.1 ma
285 C	polished	.30 V	.16 ma	.25 V	2.0 ma
272 D	unpolished	.34 V	.2 ma	.26 V	2.15 ma
285 D	polished	.32 V	.22 ma	.26 V	2.5 ma
271 C	unpolished	.38 V	.06 ma	.27 V	2.05 ma
286 D	polished	.34 V	.2 ma	.275 V	1.8 ma

(all readings at 100 mw/cm² irradiation)

TABLE 1

Current and Voltage of Front Wall CdS Cells on Mo

<u>Cell #</u>	<u>Size</u>	<u>First Reading</u>		<u>Date</u>	<u>Second Reading</u>		<u>Date</u>
		<u>V_{oc}</u>	<u>I_{sc}</u>		<u>V_{oc}</u>	<u>I_{sc}</u>	
10-8(2)	6.25 cm ²	.40 V	38 ma	11-30-62	.39 V	37 ma	4-29-63
10-8(1)	6 cm ²	.38 V	34 ma	11-30-62	.38 V	34 ma	4-29-63
12-11B1	1 cm ²	.43 V	13.2 ma	12-12-62	.45 V	10.1 ma	4-29-63
12-11B2	1 cm ²	.36 V	10.4 ma	12-12-62	.37 V	9.6 ma	4-29-63

TABLE 2

Current and Voltage of Back Wall CdS Before and After Storage

III. BASIC STUDIES

A. Hall Measurements

The usual open circuit voltage which is obtained with the $\text{CdS-Cu}_x\text{S}_y$ system is in the .42-.48 V range with only an occasional value of .50-.51 volts. These values are lower than those reported by others (.45-.56 v) and are considered lower than should be the case even if a $\text{CdS-Cu}_x\text{S}_y$ cell is operating as a cell of a single band gap of 1.3 ev.

Measurement of the Hall effect in semiconducting materials is the most direct method for determination of such basic conduction processes as the carrier type, concentration, and mobility. It is through the correlation of Hall measurements on the individual films (CdS and Cu_xS_y) and the open circuit voltages obtained with films having different properties that improvement, if not explanation, of the low open circuit voltages will be obtained.

Experimental Apparatus

The measurement of the Hall effect in high resistivity materials has been considered a difficult experimental problem. ⁽⁷⁾ One of the complicating factors is that the impedance of the external circuit must be much greater than that of the sample under test and the sensitivity must be in the microvolt range. All wiring in the external circuits require Teflon insulation and shielding to assure minimal leakage. It is also necessary that some compensation be made for misalignment voltages. Physical movement of a contact point to zero out misalignment voltages is not practical in materials like CdS because of the difficulty in producing the necessary ohmic characteristics in the contact. The instrumentation necessary for direct Hall measurements was not available until this quarter when we were

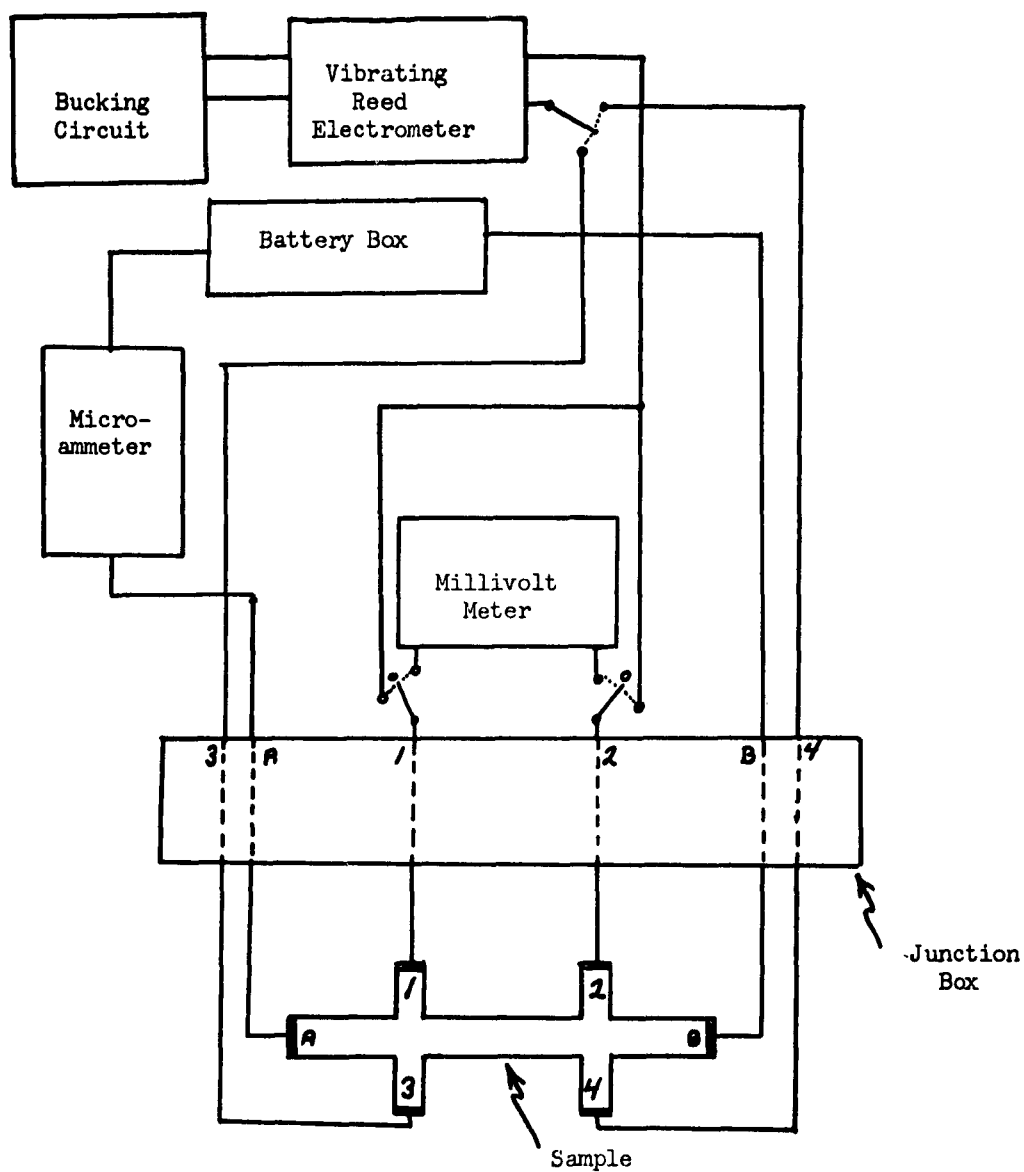


FIGURE 7
Block Diagram of Hall Effect Measurement Apparatus

able to obtain on a loan basis from ASD a Cary-type 30 vibrating reed electrometer. The open circuit impedance of this instrument can be as great as 10^{15} ohms.

The block diagram of the equipment assembly used in the measurement of the Hall effect is shown in Figure 7. The sample under test is equipped with six contact points. Two are on the end of the sample and the other four are divided with two on either side and aligned so that contacts 1 and 2 are directly across from 3 and 4 (refer to block diagram). Ohmic contact is made to the sample by ultrasonically applied indium. The resistivity is determined by measurement of the current through the sample and the potential drop between either contacts 1 and 2 or contacts 3 and 4. If the sample is uniform and properly aligned these potential drops will be the same. Hall voltage measurements are made between contacts 1 and 3 or 2 and 4. Use of both Hall contact pairs makes possible the compensation for inhomogeneity in the sample. The Hall voltage is read directly by means of the vibrating reed electrometer. Due to possible misalignment of the Hall probes there is a potential present even when there is no magnetic field. A bucking circuit was built and employed to introduce a potential in the feedback loop of the electrometer and thus reduce the misalignment voltage to zero. The bucking network is necessary because often the Hall potential will be small when compared to the misalignment voltage and thus would make direct reading difficult. (8)

The thickness (d) of thin film samples is determined by Tolansky multiple-beam interference techniques. (9)

A magnetic field as great as 20 kilogauss is supplied by a Varian Electromagnet Model V-4004-4".

Results

Initial measurements made using the apparatus described above were on single crystals of CdS. The conductivity of these crystals ranged from $0.25 \text{ ohm}^{-1} \text{ cm}^{-1}$ to $1.5 \times 10^{-3} \text{ ohm}^{-1} \text{ cm}^{-1}$, the corresponding carrier concentrations range from $4.4 \times 10^{15}/\text{cm}^3$ to $6 \times 10^{13}/\text{cm}^3$ and the mobilities range from 150 to 335.

Mobility and carrier concentration determinations in thin films of CdS are shown in the table below:

TABLE 3
Hall Measurements

Sample Type	Mobility μ $\text{volt}^{-1} \text{ cm}^{-1} \text{ sec}^{-1}$	Hall Coefficient R_H	Conductivity $\text{ohm}^{-1} \text{ cm}^{-1}$	Carrier Concentration carriers/cc
PH-887E (CdS Film)	18.2	-2700	6.85×10^{-3}	2.3×10^{15}
PH-887B (CdS Film)	22.5	-1030	2.14×10^{-2}	6.2×10^{15}
PH-887M (CdS Film)	95.5	- 955	0.10	6.5×10^{15}
PH-887N (CdS Film)	82.0	2340	3.5×10^{-2}	2.67×10^{15}

From these results, which must be regarded as preliminary, it appears that the variation in conductivity of the CdS thin films is due for the greater part to the differences in mobility rather than the carrier concentration.

Attempts to make measurement of the Hall voltage in thin film Cu_xS_y have not proven successful. Calculations based on mobilities reported in the literature ⁽¹⁰⁾ show that a Hall voltage of about 10 to 40 microvolts may be expected but due to the high conductivity of the material

this potential is very difficult to detect. Estimation of the carrier concentration ($10^{21}/\text{cm}^3$) in the p-type Cu_xS_y films was made by comparing the film to a p-type silicon sample of known carrier concentration using the thermoelectric probe method.

B. Barrier Formation

It was suggested in the Technical Report, Part I⁽¹¹⁾ covering the first 12 months effort that a discrete heterojunction was being formed between an n-type CdS film and a p-type Cu_xS_y film. This suggestion was supported by the unique spectral response of a Cu_xS_y -CdS-CdSe cell and that of a Cu_xS_y -CdSe cell. Also it was pointed out that all photovoltaic response was lost if the Cu_xS_y layer was removed either by etching or abrasion. Additional evidence of the existence of a heterojunction is indicated in the comparison of the two spectral response curves (Figure 8) of the front and back wall response of the same Cu_xS_y -CdS photovoltaic cell.

It can be seen that the peaks ($\sim 490 \text{ m}\mu$ and $\sim 560 \text{ m}\mu$) shift in opposite directions to each other as the irradiation is changed from front wall (energy incident upon the Cu_xS_y) to back wall (energy incident upon the CdS). This shifting is as would be predicted if it is assumed that the usable electron-hole pairs are being generated very close to each side of the junction and if the filtering action of the film materials on energy on greater than the band gap is considered.

The possibility is being studied of using single crystal slices of n-type materials to improve the copper sulfide film deposition techniques without the interfering variable of thin film surface as the substrate for the Cu_xS_y .

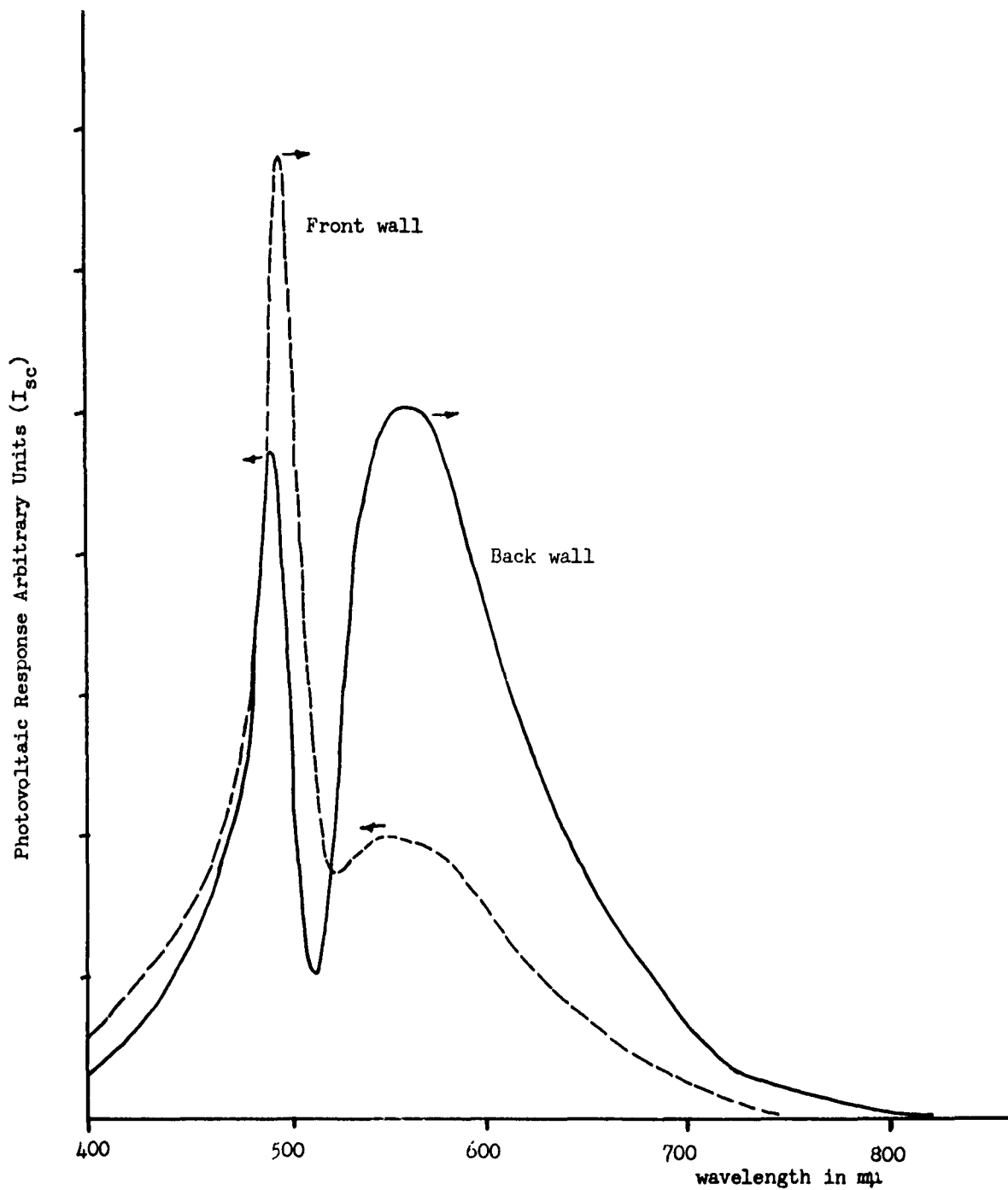


FIGURE 8

Spectral Response of a Cu_xS_y -CdS Thin Film Cell

To date single crystals of CdS, CdTe, and Si have been used as substrates for the chemical spray deposition of thin films (.05-.09 μ) of Cu_xS_y . The results of these preliminary depositions are tabulated in Table 4 given below:

TABLE 4
Results of Junction Formation with Single Crystals

<u>Single Crystal Substrate</u>	<u>Resistivity</u>	<u>Film Barrier</u>	<u>V_{oc}</u>
CdS	665 ohm-cm	Cu_xS_y	.44
CdS	166 ohm-cm	Cu_xS_y	.44
CdS	4.18 ohm-cm	Cu_xS_y	.44
CdS	5.35 ohm-cm	Cu_xS_y	.45
CdTe	unknown	Cu_xS_y	.15
Si	1 ohm-cm	Cu_xS_y	.24
Si	200 ohm-cm	Cu_xS_y	.35

As can be seen, no attempts were made to measure efficiencies but the spectral responses were measured of cells made with each type of single crystal. These spectral responses are shown in Figure 9, 10, and 11. The spectral response of Cu_xS_y on single crystal CdS is typical of what is found with Cu_xS_y -CdS (film) cells, but the response of Cu_xS_y -CdTe is that of a back wall cell where all the incident energy greater than the band gap is being absorbed prior to reaching within diffusion length of the barrier. The response of the Cu_xS_y -Si cell is given together with the response of a typical p-on-n silicon single crystal photovoltaic cell; although the long wavelength response of the Cu_xS_y -Si cell appears greater, the most interesting feature is the possibility using this

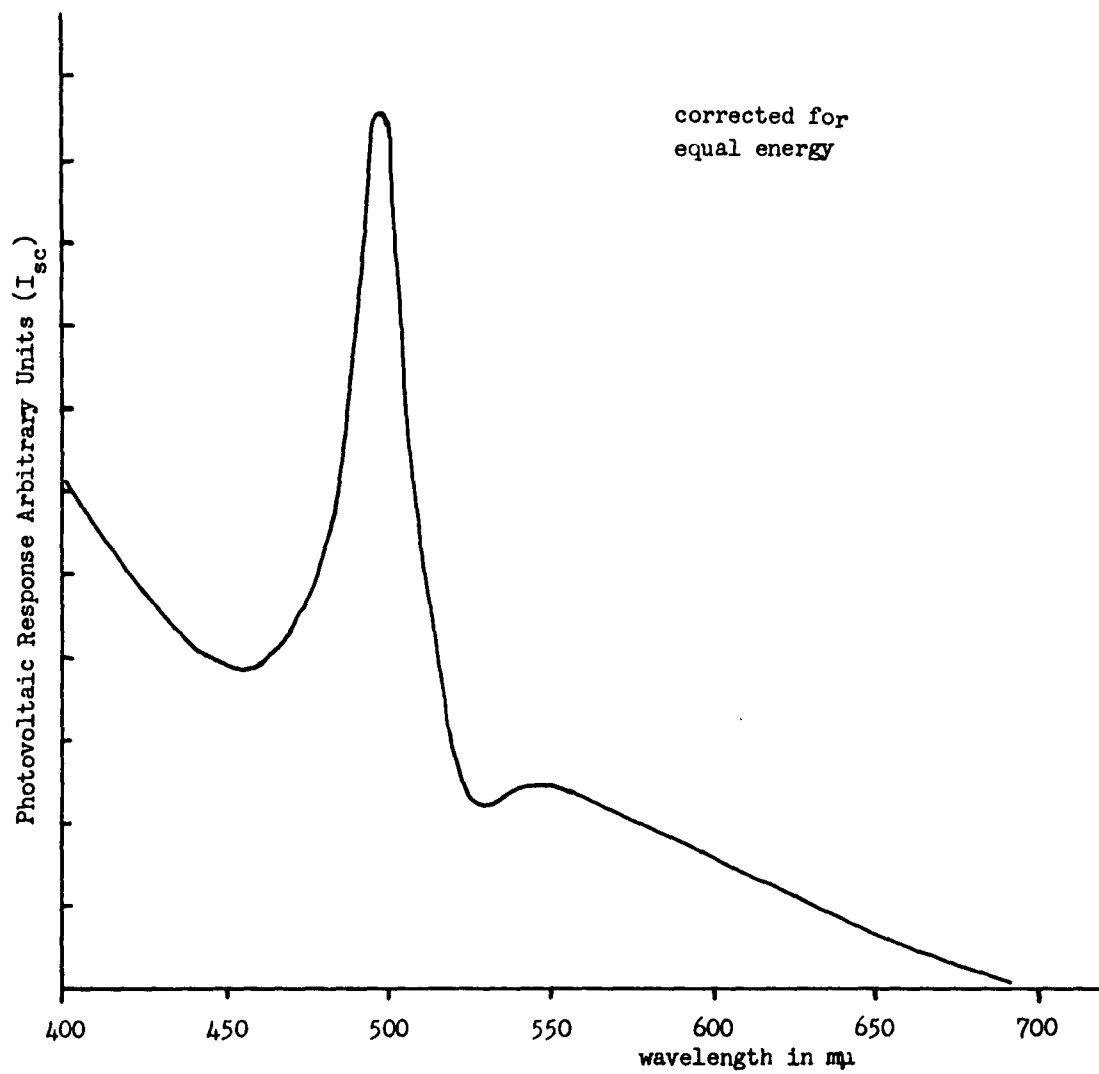


FIGURE 9
Spectral Response of a Cu_xS -CdS Single Crystal Cell

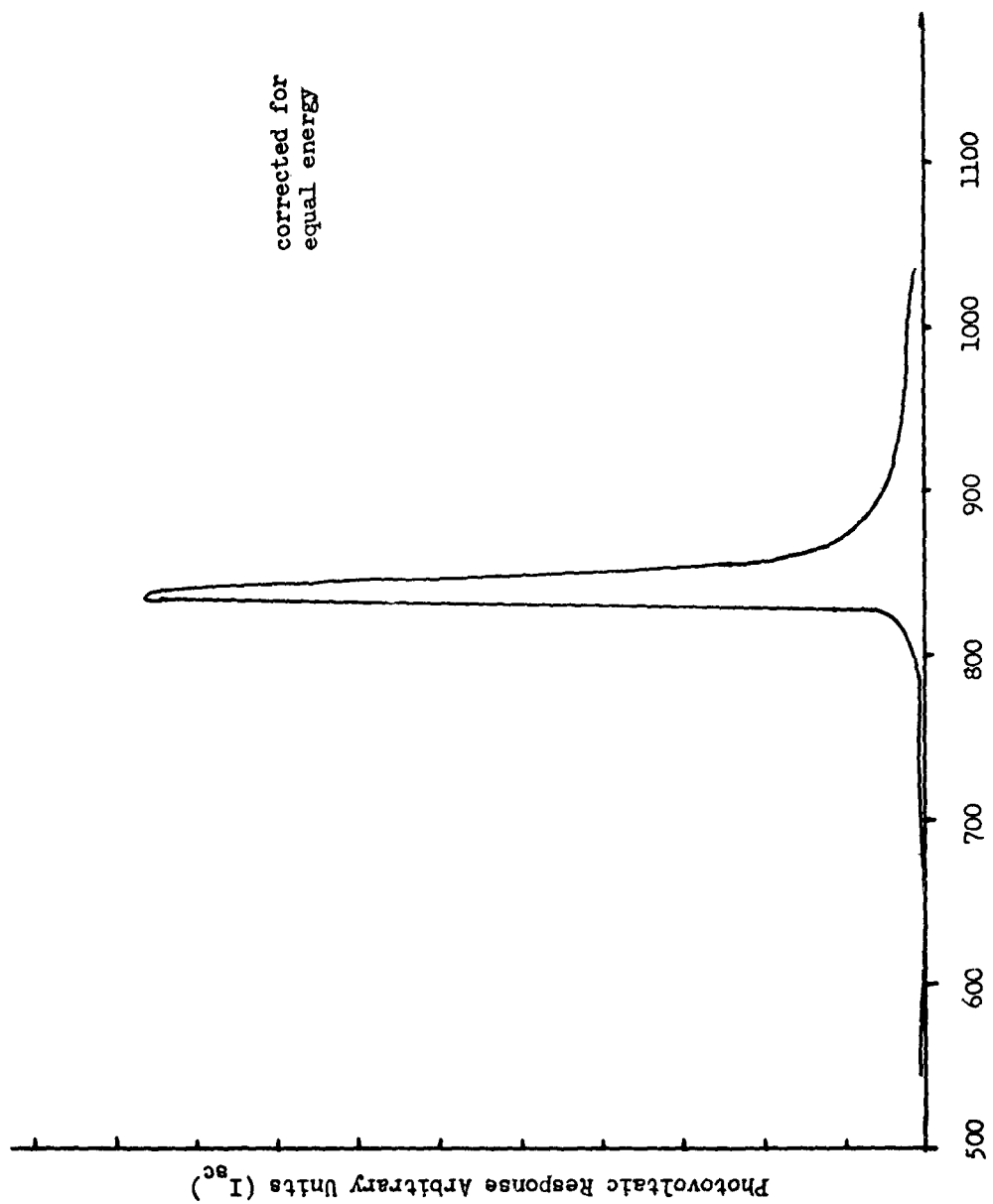


FIGURE 10
Spectral Response of a $\text{Cu}_x\text{S}_y\text{-CdTe}$ Single Crystal Cell

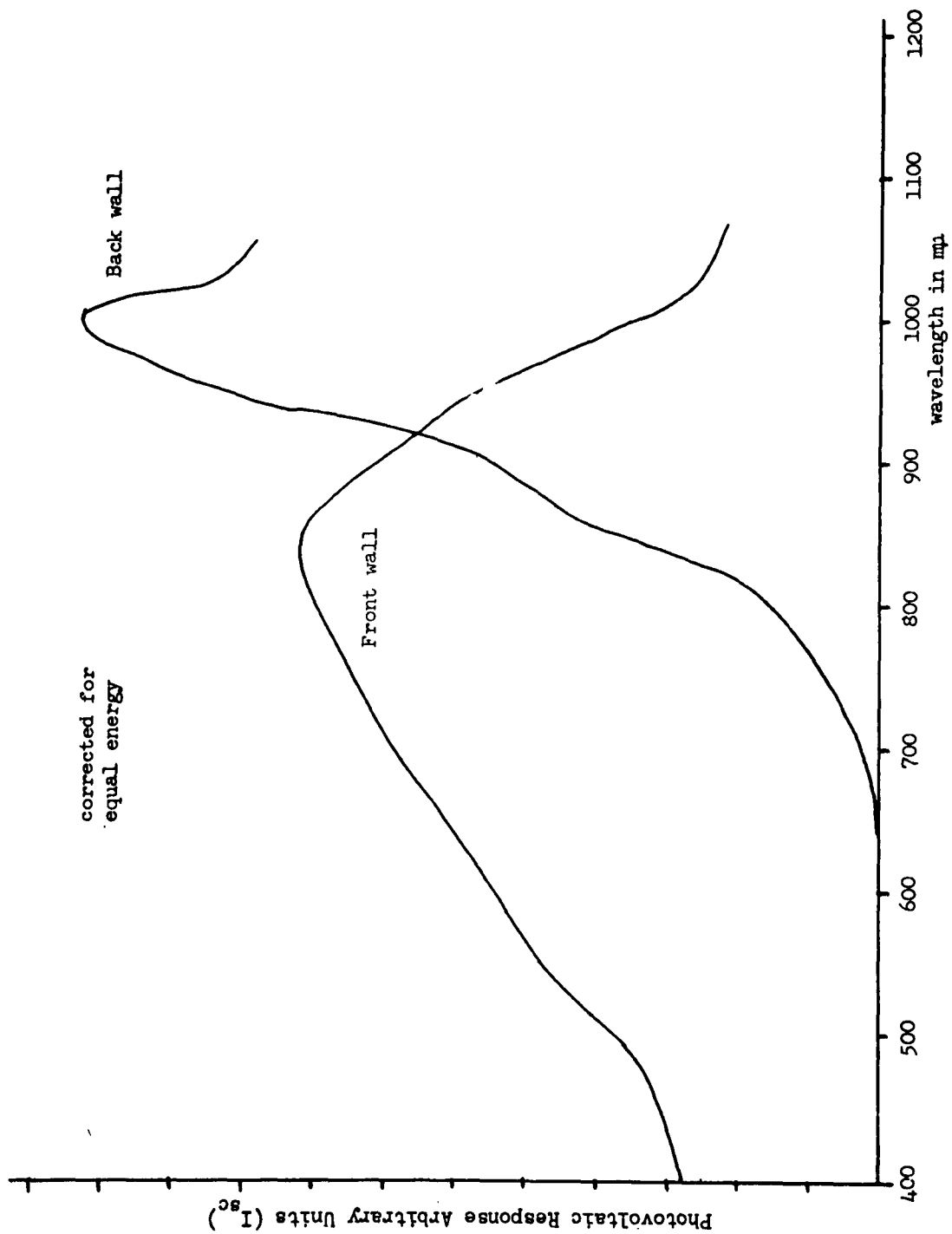


FIGURE 11
Spectral Response of a Cu_xS_y -Si Single Crystal Cell

Cu_xS_y -Si system to improve barrier forming techniques and perhaps experimentally gain an insight into the controlling factor or factors in the open circuit voltage of a heterojunction of an n-type and p-type material whose energy band gaps differ.

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